Pakistan J. Sci. Ind. Res., Vol. 17, No. 6, December 1974

MICRODETERMINATION OF PHENOL

M.H. HASHMI, S.P. HAMDANI and M. SARWAR

PCSIR Laboratories, Lahore

(Received June 6 , 1974)

Abstract. Phenol has been determined in the range of 58.5–585 μg. *N*-Bromosuccinimide is used as direct titrant and Bordeaux red as an indicator. The maximum relative standard deviation is 1.2% in the case of 58.5 μg sample. The method is simple and accurate. Resorcinol, α-naphthol and aniline interfere in this procedure.

A thorough survey of literature indicates that halogenation is the most convenient method for the estimation of phenol. The basic principle involved in these methods is that hydroxyl group present in the benzene nucleus makes substitution quite easy. Almost all the methods which are based on halogenation work on the basis that a known excess of the halogenating agent is added and then the residual amount of the reagent after it has reacted, is back-titrated. Schulek and Burger used iodine monobromide.[1] This reagent is very unstable and cannot be used as a primary standard. Its shelf-life is also very limited. The same authors used bromine water for the estimation of phenol.[2] Bromine water is highly unstable and it must be standardized before every titration. Moreover, it cannot be stored. The most convenient method of all these is bromination by bromide–bromate mixture.[3–4] This method is time consuming since back-titration is involved. Mlodecka has written a review on the subject of bromination and gave 37 references.[3] As from all these methods, none was found completely satisfactory so he attributed a number of reasons for the erroneous results. The bromine consumption depended on the working conditions, i.e. time of bromination and also the excess of bromine added.

To avoid all this confusion of time of bromination and back-titration we have devised a method where there is no back-titration and no risk of excess bromination is involved. For this purpose we have used *N*-bromosuccinimide as titrant and Bordeaux red has been used as visual indicator. *N*-Bromosuccinimide has already been used for various selective determinations.[5–12] This is the only reagent which is required in this procedure. It is a primary standard and can be shelved for a considerable period. The present method is simple, direct and less time-consuming. It has been statistically found to be precise and accurate.

Experimental

Reagents

Phenol. The solution was prepared by dissolving 5.85 mg of the redistilled reagent in distilled water and diluted to 100 ml.

N-*Bromosuccinimide.* Exactly 89.0 mg of the recrystallized compound was dissolved in distilled water and diluted to 100 ml. This is 0.01N solution of *N*-bromosuccinimide.

Sodium Bicarbonate. 4% (w/v) solution in water.

Bordeaux Red. 0.05% in water.

All chemicals used were of A.R. grade.

Procedure. An accurately measured volume (1 ml) of the test solution (phenol) was placed in a 50 ml Erlenmeyer flask. 1 ml 4% $NaHCO_3$ followed by 2 drops of Bordeaux red was added. The resulting solution was then titrated against the standard 0.01N. *N*-Bromosuccinimide solution added dropwise from a microburette graduated at 0.01 ml interval. The end-point was reached when the rose red colour of the test solution changed to clear yellow. Then a blank was run for the indicator and this value was substracted from the total titer before calculations.

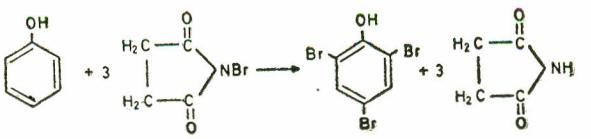
Calculations

Amount of phenol (μg) = 156.6 $(V–B)$

where 156.6 is the equivalent amount of phenol in μg/ml *N*-bromosuccinimide; V, volume (ml) of *N*-bromosuccinimide used for the titer, and B, blank (ml).

Results and Discussion

The determination of phenol with *N*-bromosuccinimide has been shown in Table 1. It can also be seen that phenol in μg quantities can be determined with *N*-bromosuccinimide accurately. Higher concentrations than these were tried but the end-point was not clear. This was due to the formation of fluffy precipitate of tribromophenol which tend to overlap the yellow endpoint. *N*-Bromosuccinimide brominates phenol to give tribromophenol. The reaction is shown in the following equation:



The equivalent weight of phenol was calculated taking above equation into consideration. This is a preferential substitution of bromine into phenol. Bordeaux red stays unattacked until phenol is consumed. A large amount of phenol was treated with *N*-bromosuccinimide under the similar condition

TABLE 1. MICRODETERMINATION OF PHENOL.

Phenol		Standard deviation (%)
Taken (μg)	found* (μg)	
58.5	56.3	1.2
117.0	118.7	1.1
234.0	233.0	0.6
351.0	254.0	0.3
468.0	470.0	0.2
585.0	585.0	0.2

*Mean of 5 titrations.

and the product separated. It was identified by IR, bromine determination and the melting point is thereof to be tribromophenol. Although NBS is a potential oxidizing agent, it does not oxidize phenol under experimental conditions. It preferentially brominates phenol.

Interfering Substances. In this procedure resorcinol, α-naphthol and aniline interfere. The above described method is of value where quick estimation of pure phenol is required.

References

1. E. Schulek and K. Burger, Talanta, **1,** 147 (1958).
2. E. Schulek and K. Burger, Ann. Univ. Sci., Budapest Rolando Eotvos Nominatae, Sect. Chim., **2,** 537 (1960).
3. J. Mlodecka, Rev. Chim., **10,** 343 (1959).
4. E. Pappa and L. Szekeres, Magy Kem. Lapja, **17,** 424 (1962).
5. M. Sarwar and K. Butt, Mikrochim. Acta., 679 (1973).
6. M. Sarwar, M. Elahi, M. Anwar and M. Hanif, Mikrochim. Acta., 846 (1970).
7. M. Sarwar, A.K. Rana and S.P. Hamdani, Microchem. J., **16,** 184 (1971).
8. M. Sarwar, S.P. Hamdani and A. Iftikhar, Mikrochim. Acta., 573 (1971).
9. M. Sarwar and R.J. Thibert, Anal, Lett., **1,** 381 (1968).
10. R.J. Thibert and M. Sarwar, Mikrochim. Acta., 259 (1969).
11. R.J. Thibert and M. Sarwar, Microchem. J., **14,** 271 (1969).
12. R.J. Thibert and M. Sarwar, Mikrochim. Acta., 615 (1969).
13. M.H. Hashmi, M. Sarwar and A. Rashid, Pakistan J. Sci. Ind. Res., **16,** 11 (1964).